Acta Cryst. (1951). **4**, 502

Optical Demonstration of Electron-Diffraction Effects due to Small Particle Size

By W. G. Burgers

Technical University, Laboratory for Physical Chemistry, Delft, Holland

(*Received* 3 *April* 1951)

1. In a recent paper by Rees & Spink (1950), 'The shape transform in electron diffraction by small crystals', photographs are reproduced showing the reflexions on the $11\bar{2}0$ ring of zinc oxide (ZnO). They show clearly that each reflexion on the ring is accompanied by a number of subsidiary maxima, which extend in a radial direction, perpendicular to the ring circumference.

As set forth in the paper, these maxima are due to the fact that the individual crystallites are thin needles of diameter 100–500 A., considerably elongated in the direction of the hexagonal axis.† 'The shape transform of such crystals in reciprocal space will therefore extend appreciably only in the reciprocal-lattice plane normal to c^*, and the resulting diffractions will be streaked at angles determined by the orientation of the crystal with respect to the beam and the indices of the particular reflexion.'

Similar effects in ZnO photographs were observed by Hillier & Baker (1946) and are also visible in the accompanying photograph of ZnO‡ (Fig. 1 (*a*)). It shows the elongation of the individual spots (the division in subsidiary maxima is not so well visible), and moreover the *varying* direction of the striae with regard to the corresponding diffraction circle. If we restrict ourselves to the innermost circles with indices $(10\bar{1}0)$, (0002), $(10\bar{1}1)$, $(10\bar{1}2)$ and $(11\bar{2}0)$, we see that the individual striae have directions respectively *perpendicular* [for $(10\bar{1}0)$ and $(11\bar{2}0)$], *parallel* [for (0002)] and *inclined* [for $(10\bar{1}1)$ and $(10\bar{1}2)$] to the diffraction circles. This is indicated once again in a schematic way in the accompanying drawing (Fig. 1 (*b*)).

2. This remarkable effect can be illustrated in a 'qualitative' way by means of the apparatus for optical demonstration of electron-diffraction photographs, as constructed several years ago by the author in collaboration with Ploos van Amstel (Burgers & van Amstel, 1936). With its help the understanding of complicated geometrical features of diffraction photographs can often be facilitated.

The principle of the demonstration is based on the fact that the pattern of the electron-diffraction photograph practically conforms to the 'intersection' of the reciprocal space and a plane perpendicular to the incident electron beam (this plane, for short-wave electrons, being the degeneration of Ewald's sphere of reflexion). If, therefore, we make a 'model' of the reciprocal space with lattice regions (shape transforms) of some light-reflecting material and we illuminate this model by a 'plane of light' in a direction corresponding to the direction of the incident electron beam with regard to the crystalline preparation under investigation, those parts of the shape transforms which are illuminated form a pattern closely resembling the actual diffraction pattern. Examples are shown in the paper referred to above (see also Trillat, 1939).

3. In the present case the electron-microscope image showed thin needles of ZnO, depending from the edge of a specimen holder. As quoted above from Rees & Spink's paper, the reciprocal-lattice regions extend in this case only perpendicular to the c^* axis and can to a first approximation be considered as flat disks. We thus made a 'reciprocal lattice' as shown in Fig. 3, at *L*, consisting of supporting rods, representing the a^* and the (double of the) c^* axes of the ZnO reciprocal lattice.† At their ends, and also in the 'diagonal' corners, disks of transparent plastic about 3 cm. diameter were fixed perpendicular to the c^* axis, representing the reciprocal-lattice domains. The model shown contains therefore the domains (0002), $(000\bar{2})$; $(10\bar{1}0)$, $(\bar{1}0\bar{1}0)$; $(10\bar{1}2)$, $(10\bar{1}\bar{2})$, $(\bar{1}0\bar{1}2)$ and $(\bar{1}0\bar{1}\bar{2})$.

Owing to the small focusing depth of the electron microscope, together with the fact that the thickness of the needles in the direction of the electron beam, and therefore their absorption power, increased for needles in 'oblique' positions, the diffraction pattern may be assumed to be mainly due to those needles which are perpendicular to the incident beam.

The diffraction effect, which would be obtained if a single ZnO crystallite were exposed to an electron beam perpendicular to its *c* axis, corresponds in the demon-

---

† Apart from the effect due to the particular shape transform, the photographs show other maxima due to refraction effects; these latter are not considered in the present note.

‡ For this photograph and for particulars regarding the conditions under which it was taken (see text, § 3) I am indebted to Ir A. C. van Dorsten from the Philips Scientific Laboratory at Eindhoven, who obtained it some time ago with the Philips electron microscope.

† As $|a^*| = 1/\frac{1}{2}a\sqrt{3}$ and $|c^*| = 1/c$, then, with $a = 3{\cdot}2$ and $c = 5{\cdot}2$ A., and taking $(1\text{ A.})^{-1} = 20$ cm., the rods supporting the disks '$10\bar{1}0$' and '0002' ought to have lengths of 7·2 and 7·7 cm. respectively. When constructing the model on this scale, it appeared that the pattern was too compact to show the effect clearly. We therefore increased the length of the 0002 rod to 9 cm. and omitted the $(10\bar{1}1)$ disk. Owing to this, the model shows only *one* (intermediate) line with 'oblique' reflexions instead of two.

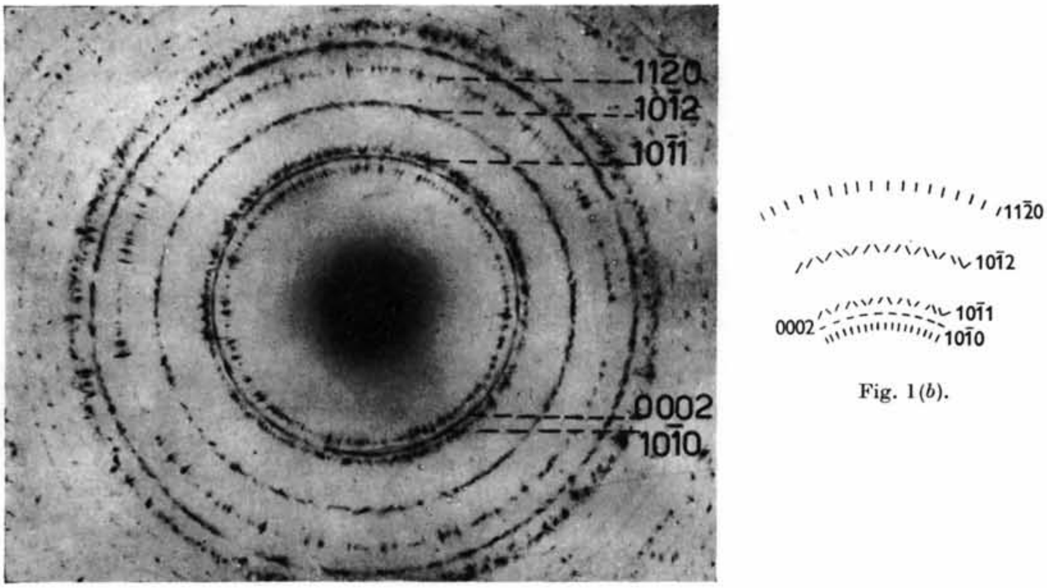

Fig. 1(*a*).

Fig. 1(*b*).

Fig. 1. Electron-diffraction photograph of ZnO showing the varying direction of individual striae with respect to corresponding diffraction rings (cf. drawing).

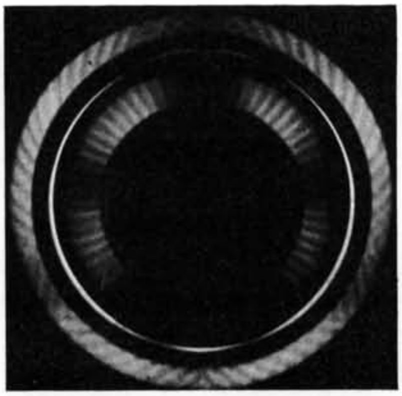

Fig. 2. Photograph of the 'diffraction pattern', as shown by the demonstration apparatus in a darkened room.

stration apparatus to an irradiation of the model by the 'plane of light' D (produced by the lamp A inside the box B and the vertical slit C). The other orientations of the crystallites perpendicular to the incident beam can be obtained starting from this arbitrarily chosen orientation by (a) a rotation about the c^* axis, (b) a rotation of the whole set of orientations so obtained about an axis perpendicular to the c^* axis, i.e. parallel to the direction of the incident electron beam.

The first rotation leaves the 'disk' (0002) in its original position and moves the other disks *outside* the 'plane of reflexion'; it is therefore immaterial and need not be realized.† The second rotation can be achieved by means of the motor M, which causes the model to rotate about the axis N, parallel to the electron beam. If now we use for lamp A a sodium or mercury gas-discharge lamp,‡ we obtain the stroboscopic effect shown in Fig. 2, which is a photograph of the actual pattern visible in a darkened room. The demonstration illustrates in a rather striking way the difference in direction of the individual diffraction striae, namely, perpendicular, parallel and oblique to the diffraction circles, as it has to be on the basis of the actual shape transform in reciprocal space.

The resemblance with the electron-diffraction photograph, however, cannot be expected to be more than 'qualitative', as diffraction by needles in an *oblique* position with regard to the incident beam, although not giving rise to (0002) reflexions on account of the small value of the glancing angle θ, might give reflexions such as $(10\bar{1}0)$, for example. These reflexions would show shapes corresponding to a different 'cross-section' of the 'plane of reflexion' with the disk-shaped shape transforms. Actually, the $(10\bar{1}0)$ diffraction ring shows, apart from striae perpendicular to the circumference of the ring, also more 'roundish' (disk-shaped) spots.

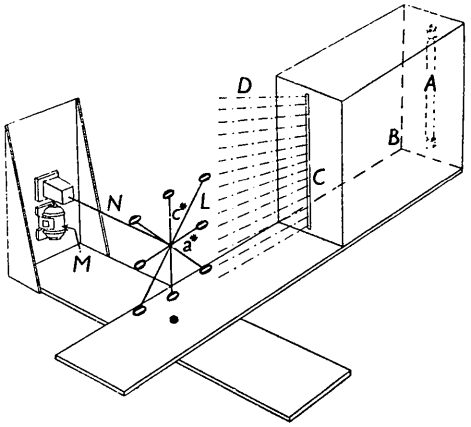

Fig. 3. Demonstration apparatus with reciprocal-lattice model of ZnO. A, sodium gas-discharge lamp; B, metal box; C, slit; L, reciprocal-lattice model; M, motor; N, axis of rotation parallel to direction of incident electron beam.

References

Burgers, W. G. & Amstel, J. J. A. Ploos van (1936). *Z. Krystallogr.* A, **95**, 54.

Hillier, J. & Baker, R. F. (1946). *J. Appl. Phys.* **17**, 12.

Rees, A. L. G. & Spink, J. A. (1950). *Acta Cryst.* **3**, 316.

Trillat, J. J. (1939). *J. Phys. Radium*, **10**, 465.

---

† This rotation would, however, bring the $(11\bar{2}0)$ domain, discussed in Rees & Spink's paper, into the 'plane of reflexion' and show the 'radial' direction of the corresponding interference striae. As this would require an extension of the model in the direction of the b^* axis, which would make it more 'voluminous', we have left this out, as the same effect could be shown more easily for the $(10\bar{1}0)$ reflexion.

‡ I am indebted to Dr F. J. Lebbink for this suggestion.

Acta Cryst. (1951). **4**, 503

Pseudo-Cubic Alkaline-Earth Tungstates and Molybdates of the R_3MX_6 Type*

By E. G. Steward and H. P. Rooksby

Research Laboratories of The General Electric Company Limited, Wembley, England

(*Received* 28 *February* 1951)

The formation of R_3WO_6-type alkaline-earth tungstates containing one or more alkaline-earth metals is described. They have structures approaching, and in some examples attaining, cubic symmetry (space group $Fm3m–O_h^5$). A structure, suggested by analogy with $(NH_4)_3FeF_6$, is tested and found to be correct in representing this arrangement.

The deformations from true cubic symmetry exhibited by the compounds are briefly discussed, and examples are given of the temperature dependence of this deformation.

Molybdates of the same type may be formed.

Introduction

Many tungstates of the type RWO_4, where R is an alkaline earth or other divalent element, are well-established chemical compounds. The crystal structures of these alkaline-earth compounds belong to two main classes. Calcium, strontium and barium tungstates are tetragonal, with space group $I4_1/a–C_{4h}^6$, whilst magnesium tungstate is a member of a class, including

---

* Communication No. 470 from the Staff of the Research Laboratories of The General Electric Company Limited, Wembley, England.